Powder coating

Patent Number:	EP0687714
Publication date:	1995-12-20
Inventor(s):	LIBKE SHANNON (JP); TSUTSUI KOICH (JP); UENO TASABURO (JP); KENNY JOHN (JP)
Applicant(s)::	NIPPON PAINT CO LTD (JP)
Requested Patent:	EP0687714, B1
Application Number:	EP19950108012 19950524
Priority Number(s):	JP19940115589 19940527
IPC Classification:	C09D5/03
EC Classification:	C09D5/03B
Equivalents:	DE69509440D, DE69509440T

Abstract

The present invention relates to a powder coating improved in transportability and capable of yielding a film of excellent appearance. Powder coating of the present invention comprises a film forming resin having a volume average particle diameter of 20-50 mu m with a standard deviation of particle diameter distribution not greater than 20 mu m.

(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 687 714 A1**

(12) **EUROPEAN PATENT APPLICATION**

(21) Application number: **95108012.6**

(51) Int. Cl.[6]: **C09D 5/03**

(22) Date of filing: **24.05.95**

(30) Priority: **27.05.94 JP 115589/94**

(43) Date of publication of application:
20.12.95 Bulletin 95/51

(84) Designated Contracting States:
DE FR GB

(71) Applicant: **Nippon Paint Co., Ltd.**
1-2 Oyodokita 2-chome
Kita-ku,
Osaka-shi,
Osaka 531 (JP)

(72) Inventor: **Tsutsui, Koich**
13-4, Osumigaoka 5-chome,
Tanabe-cho
Tsuzuki-gun,
Kyoto 610-03 (JP)
Inventor: **Ueno, Tasaburo**
8-11, Nishino
Sakai-shi,
Osaka 588 (JP)
Inventor: **Kenny, John**
3-20-302, Korikitanomachi
Neyagawa-shi,
Osaka 572 (JP)
Inventor: **Libke, Shannon**
2-7-301, Korienyamanotecho
Hirakata-shi,
Osaka 573 (JP)

(74) Representative: **Brehm, Hans-Peter, Dr.,**
Dipl.-Chem.
Patentanwälte
Kern, Brehm & Partner
Albert-Rosshaupter-Strasse 73
D-81369 München (DE)

(54) **Powder coating**

(57) The present invention relates to a powder coating improved in transportability and capable of yielding a film of excellent appearance. Powder coating of the present invention comprises a film forming resin having a volume average particle diameter of 20-50 µm with a standard deviation of particle diameter distribution not greater than 20 µm.

TECHNICAL FIELD

The present invention relates to a powder coating which is improved in transportability and capable of yielding a film of improved appearance.

PRIOR ART

A powder coating generally comprises a film-forming resin having a volume average particle diameter of about 30-40 μm and capable of yielding a film on application to a substrate surface by an electrostatic spray coating, fluidized-dipping or other coating technique and subsequent baking.

However, such a powder coating does not form a glossy, smooth film so that it has the disadvantage of poor appearance.

As a technology for overcoming the above-mentioned disadvantage and providing a film of improved appearance, Japanese Patent (JP) Application H-3-264025 discloses a powder coating having an average particle diameter smaller than the usual range and, yet, with the maximum proportion of small particles being limited, namely a powder coating having an average particle diameter of 5-20 μm and a proportion of particles not exceeding 5 μm in diameter accounting for 25 weight % or less.

This powder coating yields a glossy, dense and smooth film but since its average particle diameter is smaller than that of the earlier powder coating, it is poor in transportability so that it tends to plug the pipeline for pneumatic or other transport.

Under the circumstances the object of the present invention is to provide a powder coating which can form a film of good appearance and have excellent transportability.

SUMMARY OF THE INVENTION

The gist of the present invention resides in providing a powder coating comprising a film-forming resin having a volume average particle diameter of 20-50 μm with a standard deviation of particle diameter distribution not greater than 20 μm.

BRIEF DESCRIPTION OF THE DRAWING

Fig. 1 shows a schematic diagram of the optics of the apparatus used for the evaluation of film appearance.

DETAILED DESCRIPTION OF THE INVENTION

The volume average particle diameter of the powder coating of the present invention is 20-50 μm. If the volume average particle diameter is less than 20 μm, the powder tends to plug the pipeline of the pneumatic transport system line to cause a transportation trouble, while exceeding 50 μm fails to provide for a film of good appearance. The preferred range is 20-40 μm. The value of volume average particle diameter can be measured with a particle size analyzer of electromagnetic wave scattering, such as Leeds & Northrop's Microtrac-II.

The standard deviation of particle diameter distribution of the powder coating of the present invention is not greater than 20 μm. If the standard deviation exceeds 20 μm, a film of satisfactory appearance will not be obtained. For better results, the standard deviation of particle diameter distribution is preferably not greater than 16 μm and, preferably, not greater than 13 μm. When the standard deviation is not greater than 16 μm, the film has a satisfactory appearance and when it is not greater than 13 μm, a still more satisfactory film appearance is obtained.

The standard deviation of particle diameter distribution of a powder coating can be calculated from the data obtained with the particle size analyzer by means of the following equation.

$$\sigma = [\Sigma \{ (D - X)^2 F) / \Sigma F]^{1/2}$$

wherein σ represents the standard deviation of particle diameter distribution. D represents the particle diameter of each individual particle. X represents the volume average particle diameter as expressed by $X = \Sigma (DF) / \Sigma E$, where F represents the frequency of the particle.

The diameter of the largest particle in the powder coating of the present invention is not greater than 90 μm. If there are particles exceeding 90 μm in diameter, a film of good appearance cannot be obtained. The

preferred diameter of the largest particle is not less than 80 μm.

In the preferred powder coating of the present invention, the diameter of the largest of particles constituting the coating is not greater than 90 μm and the diameter of the smallest of particles is not less than 1 μm. The presence of particles smaller than 1 μm results in poor transportability.

The powder coating of the present invention comprises a film-forming resin of binder component. The film forming resin is that commonly used in the field of powder coating , but not limited in its kind. For example, both thermoplastic resin and thermosetting resin can be mentioned.

The type of thermoplastic resin that can be used is not particularly limited. Thus, vinyl resins such as poly(vinyl chloride), polyethylene resin, polyamide resin, etc. can be employed with particular advantage.

The type of thermosetting resin that can be used is not particularly limited, either. Thus, epoxy resin, thermosetting acrylic resin, and thermosetting polyester resin, among others, can be employed among others.

When a thermosetting resin is used as the film-forming resin, the powder coating of the present invention preferably contains a curing agent and a curing accelerator.

The amount of said curing agent is preferably in the range of 5-80 parts by weight based on 100 parts by weight of the thermosetting resin used as the film-forming resin. If the proportion of the curing agent is less than 5 parts by weight, the hardening effect will be insufficient. On the other hand, if it exceeds 80 parts by weight, the hardening will proceed too far, so that the physical properties of the film will be adversely affected.

The amount of said curing accelerator is preferably 0.1-5 parts by weight based on 100 parts by weight of the thermosetting resin used as the film-forming resin. If the proportion of the curing accelerator is less than 0.1 part by weight, the curing of the resin will be sufficient. If it exceeds 5 parts by weight, the hardening will proceed too far so that the physical properties of the film will be adversely affected.

When an epoxy resin is used as the film-forming resin in accordance with the present invention, a curing agent and/or a curing accelerator, e.g. phthalic anhydride, an amine compound, an imidazole compound, dicyandiamide, etc., and/or a different resin such as acrylic resin can be concomitantly used where necessary.

When a thermosetting acrylic resin is used as said film-forming resin in the practice of the present invention, other resins such as epoxy resin, melamine-formaldehyde resin, etc., and curing agents such as polybasic carboxylic acids, blocked isocyanates, etc., can be selectively used where necessary.

When a thermosetting polyester resin is used as said film-forming resin in the practice of the present invention, other resins such as melamine-formaldehyde resin, epoxy resin, etc. and curing agents such as polybasic acids, blocked isocyanates, triglycidyl isocyanurate, etc. can be selectively employed where necessary.

The powder coating of the present invention may contain a pigment and other additives where necessary.

The additives mentioned above may be other resins, curing agents, curing accelerators or curing catalysts, surface modifiers, plasticizers, ultraviolet absorbers, antioxidants, antifoaming agents and pigment dispersants, among others.

The type of pigment that can be used is not particularly limited, thus including titanium dioxide, red iron oxide, yellow iron oxide, carbon black, phthalocyanine blue, phthalocyanine green, quinacridone red, etc., to mention just a few preferred examples.

The preferred amount of said pigment is 1-80 parts by weight based on 100 parts by weight of the powder coating. If the amount of the pigment is less than 1 part by weight, no sufficient coloring effect and other pigment effect can be obtained. If it exceeds 80 parts by weight, a film of satisfactory appearance will not be obtained.

The above-mentioned pigment and other additives may occur within the powder coating particle together with the film-forming resin, or may be present as different particles of the film-forming resin.

The powder coating of the present invention may contain any pigment that does not decrease its clarity, whether in quantity or in kind. If desired, the powder coating may not contain a pigment at all to be a clear coating capable of producing a transparent film.

The powder coating of the present invention typically comprises composite particles each consisting of a first particle containing part of a film-forming resin and a second particle disposed on the surface of said first particle, said second particle containing the remainder of said film-forming resin and having a glass transition temperature of 50-150 °C . The powder coating composite particle mentioned above may be such that said second particle is merely attached to the surface of said first particle or such that said second particle is partially embedded in the surface layer of said first particle. The powder coating of the present invention may optionally comprise both of these two kinds of composite particles.

As the second particle is disposed on the surface of the first particle to provide a composite particle in this manner, the first particles of the composite particles are substantially prevented from contacting each other directly so that they do not cause blocking during storage easily and the free-flowing property of the powder coating during transport is improved. Moreover, by this integration of the second particle with the surface of the first particle to provide a composite particle, a film-forming resin with a low glass transition temperature (Tg), for example a resin with Tg = 40 °C , for the first particle is able to be used.

Of the above powder coating composite particle, the second particle melts together with the film-forming resin of the first particle in the stage of baking the powder coating to form a finished film, with the result that the film is free of defects such as surface irregularities due to the presence of the second particle.

The second particle mentioned above is preferably made of the same resin as the film-forming resin described above. For the production and other practical reasons, vinyl resin, acrylic resin, epoxy resin, polyester resin, melamine resin, etc. are generally selected. Among them, vinyl resin is preferred from the standpoint of ease of production and freedom in design.

The resin constituting the second particle has a Tg value of 50-150 °C . If the Tg is less than 50 °C , the effect of grafting the second particle onto the first particle may not be realized. If the Tg is over 150 °C , the effect will not be further improved. The preferred Tg range is 70-120 °C .

The volume average particle diameter for the second particle is preferably smaller than the volume average particle diameter of the powder coating. The volume average particle diameter for the second particle is preferably 0.001-10 μm and, for still better results, 0.01-5 μm.

The proportion of said second particle is preferably 0.05-35 parts by weight relative to 100 parts by weight of the powder coating. If it is less than 0.05 part by weight, the effect of the second particle may not be sufficiently realized. On the other hand, if it exceeds 35 parts by weight, the appearance of the film will be adversely affected.

The second particle can be directly produced by such production techniques as emulsion polymerization, suspension polymerization, etc. As an alternative, the second particle can be obtained by producing a resin by a production technique such as solution polymerization, block polymerization or the like, pulverizing the resin, and classifying the resulting powder.

The production technology that can be used for the production of the powder coating of the present invention is not particularly restricted, thus including the various processes conventionally used for the manufacture of powder coatings. A typical process may comprise mixing said film-forming resin, optional pigment, and other additives in a Henschel mixer, super mixer, ball-mill, Bumbery mixer or the like, feeding the resulting uniform mixture to an extruder, hot roll or other compounding machine where it is melt-compounded to provide a uniform dispersion of the additives in the molten film-forming resin and broken into chips. The chips are then crushed with an impact mill, such as a hammer mill, or a pneumatic mill such as a jet mill, and the resulting powder is classified to provide the powder coating of the present invention.

The above classification accomplishes the above-mentioned particle size distribution, with the proportion of particles larger than the above-defined particle diameter range and of particles smaller than the range being both diminished. The above classification can be carried out using a Tyler standard sieve of 170 mesh, preferably 200 mesh, a fluid classifier such as a dispersion separator or a micron separator, which separates and removes particles larger than 90 μm, preferably 80 μm, or a cyclone, dispersion separator, micron separator or other fluid classifier which separates and removes particles smaller than 1 μm.

The technology for producing the powder coating of the present invention is not limited to the methods described above. A typical alternative method comprises mixing the starting materials into a solvent and drying and pulverizing the resulting mixture and pulverizing it or spray-drying the mixture to provide a powder. Where necessary, the powder can be crushed and classified.

For the production of the powder coating of the present invention comprising said composite particles, the second particle component is mixed with the first particle component. In this mixing stage, the desired mode of integration or grafting of the first particle and second particle can be selectively obtained by judicious selection of a mixer and mixing conditions. For example, when a hybridizer is used as the mixer, the resulting powder coating is such that the second particle is partially embedded in the surface layer of the classified first particle. The above mixing can be accomplished with a generally used mixer such as a super mixer, Henschel mixer, hybridizer, ball-mill or the like. By the above integration and, where necessary, subsequent classification of the resulting composite particles, the powder coating comprising the composite particles according to the present invention can be obtained.

Referring to the starting materials for use in the production of the powder coating of the present invention, all components other than the resin are comprised of particles smaller than 700 μm in diameter in

a proportion of 40-100 weight %, preferably 60-100 weight %. Starting with such powder coating materials, the chip is a uniform dispersion of the pigment and other additives in the film-forming resin so that the powder coating available on crushing the chip also has a substantially uniform composition from one particle to another. Therefore, in the film-forming process, the hardening of the resin proceeds uniformly to provide a film having very satisfactory surface smoothness and other appearance qualities.

The substrate for the powder coating of the present invention is not particularly restricted, thus including steel sheets, zinc phosphate-treated steel sheets, aluminum or aluminum alloy sheets, etc. for cars, household appliances, architectural members and general products.

The coating technology compatible with the powder coating of the present invention may comprise depositing the powder coating in a suitable thickness on a substrate surface in the manner of, for example, electrostatic spray coating, fluidized-dipping or the like and baking the powder coating so deposited. Where the resin component used is a thermosetting resin, a hardened film can thus be obtained.

EXAMPLES

The following examples further illustrate the present invention but by no means define its scope.

Example 1

Using a super mixer (manufactured by Nippon Spindle Mfg. Co.), 48 parts by weight of glycidyl group-containing solid acrylic resin (Tg = 50 °C , number average molecular weight 3,000, epoxy equivalent 350), 12 parts by weight of 1,10-decanedicarboxylic acid, 2.2 parts by weight of bisphenol A epoxy resin (YD-012, Toto Kasei Corporation), 0.1 part by weight of polysiloxane surface modifier and 0.3 part by weight of benzoin were thoroughly mixed with stirring. The mixture was then melt-kneaded in a kneader (Buss), and chilled to solidify. The solidified mass was crushed to provide chips for powder coating.

The chips for powder coating thus obtained were pulverized with an impact mill (an atomizer manufactured by Fuji Powder Co., Ltd.) and the pulverizate was passed through a 0.7 mm-mesh screen and further through a 325-mesh Tyler standard sieve (pore size 44 μm) to remove particles larger than 44 μm. Then, by means of a pneumatic classifier (DS-2, Nippon Pneumatic Industries Co., Ltd.) all the particles smaller than 2 μm were removed to provide a powder coating.

Example 2

With the target classification size set as indicated in Table 1, a powder coating was prepared in the same manner as Example 1 except that a 250-mesh Tyler standard sieve (pore size 61 μm) was used in lieu of the 325-mesh Tyler standard sieve and that particles smaller than the diameter indicated in Table 1 were removed with a pneumatic classifier.

Example 3

With the target classification size set as indicated in Table 1, a powder coating was prepared in the same manner as Example 1 except that a 250-mesh Tyler standard sieve (pore size 44 μm) was used and that particles smaller than the diameter indicated in Table 1 were removed with a pneumatic classifier.

Example 4

With the target classification size set as indicated in Table 1, a powder coating was prepared in the same manner as Example 1 except that a 250-mesh Tyler standard sieve (pore size 61 μm) was used in lieu of the 325-mesh Tyler standard sieve and that the size selection with the pneumatic classifier was omitted.

Example 5

With the target classification size set as indicated in Table 1, a powder coating was prepared in the same manner as Example 1 except that a 170-mesh Tyler standard sieve (pore size 88 μm) was used in lieu of the 325-mesh Tyler standard sieve and that particles smaller than the diameter indicated in Table 1 were removed with the pneumatic classifier.

Example 6

The acrylic powder coating obtained in Example 4 was added to a finely divided acrylic resin powder with a particle diameter of 0.03-0.05 μm and Tg = 100 °C [methyl methacrylate 87, styrene 10, methacrylic acid 3 (all parts by weight)] and the mixture was dry-mixed in a Henschel mixer for 30 seconds to provide a composite powder coating. The proportion of said finely divided powder was set at 1.0 weight % of the powder coating.

The volume average particle diameter, standard deviation of particle diameter distribution, and appearance changed little front those of the powder coating prior to addition of the finely divided acrylic resin. However, a marked improvement was realized in transportability. Moreover, when the resulting composite powder coating was stored at 30 °C for 6 months, and, then, visually inspected for blocking of powders, there was no blocking at all, indicating that the composite powder was excellent in blocking resistance. On the other hand, when the powder coating of Example 4 was similarly evaluated for blocking resistance, little conglomeration of the particles was observed.

Example 7

A commercial polyester powder coating (P-100, manufactured by Nippon Paint Co., Ltd.) was adjusted to the target classification value indicated in Table 1 and using a 200-mesh (pore size 74 μm) Tyler standard sieve, particles smaller than the cut-off particle diameter indicated in Table 1 were removed with a pneumatic classifier. Otherwise, the procedure of Example 1 was repeated to provide a powder coating.

This polyester powder coating contained 17 parts by weight of a blocked isocyanate as the curing agent with respect to 100 parts by weight of the film-forming thermosetting polyester resin and had a pigment content of 40 weight % based on the total weight of the powder coating.

Example 8

A commercial hybrid powder coating (H-100, manufactured by Nippon Paint Co., Ltd.) was set to the target classification value indicated in Table 1 and passed through a 200-mesh (pore size 74 μm) Tyler standard sieve, and particles smaller than the cut-off particle diameter indicated in Table 1 were removed with a pneumatic classifier.

Otherwise, the procedure of Example 1 was repeated to provide a powder coating.

This hybrid powder coating contained 50 parts by weight of polyester resin and 50 parts by weight of epoxy resin as film-forming resin components and had a pigment content of 40 weight % of the total weight of the powder coating.

Example 9

The powder coating starting material according to the same formulation as used in Example 1 was supplemented with 20 parts by weight of titanium dioxide (CR-50, Ishihara Sangyo Kaisha, Ltd.) and the resulting powder with particles less than 700 μm in diameter accounting for less than 40 weight % were stirred to mix using super mixer. The mixture was melt-kneaded in a cokneader and, after cooling, the resulting solid was crushed to prepare chips for a powder coating of Example 9. The chips were finely divided as in Example 1. With the target classification value set as indicated in Table 1, the powder was passed through a 250-mesh (pore size 61 μm) Tyler standard sieve, and particles smaller than the particle diameter shown in Table 1 were removed with a pneumatic classifier to provide a powder coating.

Example 10

The procedure of Example 9 was repeated except that the powder coating starting material with particles less than 700 μm in diameter accounting for more than 40 weight % was used.

Comparative Example 1

A powder coating was prepared in the same manner as Example 1 except that particles larger and smaller than the range indicated in Table 1 were removed with the pneumatic classifier.

Comparative Example 2-3

With the target classification values set as indicated in Table 1, the starting material was passed through a 150-mesh Tyler standard sieve (pore size 104 µm) in lieu of the 325-mesh Tyler standard sieve and particles smaller than the cut-off particle diameter shown in Table 1 were removed with a pneumatic classifier. Otherwise, the procedure of Example 1 was repeated to provide powder coatings.

Comparative Example 4

With the target classification value set as indicated in Table 1, a powder coating was prepared in the same manner as Example 1 except that a 170-mesh Tyler standard sieve (pore size 88 µm) was used in lieu of the 325-mesh Tyler standard sieve and particles smaller than the cut-off particle diameter shown in Table 1 were removed with a pneumatic classifier.

Comparative Example 5

A commercial polyester powder coating (P-100, manufactured by Nippon Paint Co., Ltd.) was set to the target classification value indicated in Table 1 and a powder coating was prepared in the same manner as Example 1 except that a 150-mesh (pore size 104 µm)Tyler standard sieve was used and particles smaller than the cut-off particle diameter shown in Table 1 were removed with a pneumatic classifier.

This polyester powder coating contained 17 parts by weight of a blocked isocyanate as the curing agent with respect to 100 parts by weight of the film-forming thermosetting polyester resin and had a pigment content of 40 weight % based on the total weight of the powder coating.

Comparative Example 6

A commercial hybrid powder coating (H-100, manufactured by Nippon Paint Co., Ltd.) was set to the target classification value indicated in Table 1 and a powder coating was prepared in the same manner as Example 1 except that a 170-mesh (pore size 88 µm)Tyler standard sieve and particles smaller than the cut-off particle diameter shown in Table 1 were removed with a pneumatic classifier.

This hybrid powder coating contained 50 parts by weight of polyester resin and 50 parts by weight of epoxy resin as the film-forming resin component and had a pigment content of 40 weight % based on the total weight of the powder coating.

Comparative Example 7

The powder coating starting material according to the formulation of Example 1 was supplemented with 20 parts by weight of titanium dioxide (CR-50, Ishihara Sangyo Kaisha, Ltd.) and the resulting powder with particles smaller than 700 µm in diameter accounting for less than 40 weight % was mixed with stirring in a super mixer and, then, melt-kneaded using a cokneader. After cooling, the resulting solid was crushed to provide chips for powder coating.

The chips were finely divided in the same manner as Example 1 and set to the target classification value indicated in Table 1. Then, the powder was passed through a 150-mesh (pore size 104 µm) Tyler standard sieve and particles smaller than the cut-off particle diameter shown in Table 1 were removed with a pneumatic classifier to provide a powder coating.

Evaluation

Each of the powder coatings obtained was evaluated for the following parameters. The results are shown in Table 1.

1. Determination of the particle diameter and particle diameter distribution

The particle diameter was measured with a particle diameter measuring apparatus (Microtrac-II, Leeds & Northrop).

From the particle diameter frequency data thus obtained, the volume average particle diameter and standard deviation of particle diameter distribution were calculated by means of the following equation.

$$\sigma = [\Sigma \{ (D - X)^2 F\} / \Sigma F]^{1/2}$$

(wherein σ stands for the standard deviation of particle diameter distribution, D for the particle diameter of an individual particle, X for the volume average particle diameter, $X = \Sigma (DF) / \Sigma F$ where F represents the frequency of the particle)

2. Evaluation of the quality of appearance

Each of the powder coatings prepared in Examples and Comparative Examples was evenly coated on an iron plate by electro coating method and baked at 140°C for 20 minutes to form a film. The appearance of the film was evaluated in NSIC values determined with an image sharpness tester (manufactured by Suga Testing Instruments Co.). The NSIC values were determined by Fourier spectrum analysis of the rectangular wave pattern image formed by reflection light front the film surface using the optical system illustrated in Fig. 1.

As shown in Fig. 1, the emission from a light source 1 was caused to pass through a condenser 2, a pattern 3 and a projector lens 4 and the light reflected by the coated surface of the object 5 was focused on the light-receiving plane of a photodiode array 6 to provide an image wave pattern. The light source 1 and the light-receiving plane of the photodiode array 6 were disposed at an angle of θ with respect to the coated surface of the object 5.

The NSIC value is the value obtained by standardizing the sum of square roots of the powers of the fundamental frequency ν_0 and 3-fold equivalent frequency $3\nu_0$ of the image waveform from which the baseline intensity b had been subtracted for emphasizing the shape information

$$[P(\nu_0)^{1/2} + P(3\nu_0)^{1/2}]$$

with the corresponding value for the black glass plate

$$[P(\nu_0)^{1/2} + P(3\nu_0)^{1/2}]_{B.G}$$

as follows.

$$NSIC = [\{P(\nu_0)^{1/2} + P(3\nu_0)^{1/2}\} / P(\nu_0)^{1/2} + P(3\nu_0)^{1/2}\}_{B.G}] \times 100$$

This value represents the orange peel appearance (distortion from the square mesh), in the main, of the image.

3. Evaluation of transportability

In the coating system using a powder coating, the coating is generally transported from a powder feeder (fluidized bed) through an injector to a coating gun, passing through a hose.

The powder coating was continuously transported with the above coating system for one hour and the deposits of the powder coating in the injector and hose were visually inspected to evaluate the transportability of the powder coating. The following evaluation criterial were used.

◎ :No powder deposits
O :Little powder deposits
X :Powder deposits nearly obstructing the injector or the hose

Table 1

	Powder coating	Volume average particle diameter (μm) Target	Found	Standard deviation of particle diameter distribution (μm)	Particles removed by Tyler standard sieve (μm)	Particles removed by pneumatic classifier (μm)	Appearance (NSIC) (%)	Transportability
Compar. Ex. 1	Acrylic powder coating	10	9.5	7.2	44<	30<,2>	80.0	×
Example 1		20	20.1	14.3	44<	2>	69.0	○
Example 2		20	24.1	19.8	61<	1>	60.0	○
Example 3		20	26.8	10.7	44<	20>	71.6	○
Example 4		30	30.5	17.5	61<	--	62.1	○
Compar. Ex. 2		30	26.5	25.3	104<	1>	51.4	○
Compar. Ex. 3		30	30.5	35.7	104<	1>	53.4	○
Example 5		40	40.4	16.7	88<	25>	60.0	○
Compar. Ex. 4		60	58.5	19.0	88<	35>	50.2	○
Example 6		30	30.5	17.5	61<	--	61.0	◎
Example 7	Polyester powder coating	30~40	32.1	18.0	74<	8>	40.0	○
Compar. Ex. 5			30.9	26.0	104<	4>	30.0	○
								○
Example 8	Hybrid powder coating	30~40	30.0	16.8	74<	7>	35.0	○
Compar. Ex. 6			28.8	24.0	88<	2>	28.4	○
Example 9	Acrylic powder coating	30	29.6	17.5	61<	--	50.0	○
Example 10		30	29.0	17.0	61<	--	55.0	○
Compar. Ex. 7		30	30.8	32.2	104<	1>	32.1	○

The data presented in Table 1 indicate the following.

The powder coating of Comparative Example 2 and that of Comparative Example 3 having the volume average particle diameters of 26.5 μm and 30.5 μm, respectively, both of which are corresponding to the conventional clear coating, with standard deviations of particle diameter distribution over 20 μm produced films of poor appearance. The powder coating of Comparative Example 4 had a volume average particle

diameter of more than 50 μm and, although the standard deviation of particle diameter distribution was not greater than 20 μm, produced a film of poor appearance. The powder coating of Comparative Example 1 which had a volume average particle diameter of 9.5 μm with a standard deviation of 7.2 μm, corresponding to the parameters of the powder coating proposed in the patent literature mentioned hereinbefore, was poorly transportable because the average particle diameter was less than 20 μm.

The powder coating of Examples 1-6 which had volume average particle diameters within the range of 20-50 μm with standard deviations of particle diameter distribution not exceeding 20 μm were superior in transportability and produced good-looking films with the NSIC values not less than 60%. Particularly the powder coating of Example 1 which had a standard deviation not exceeding 16 μm produced a film of excellent appearance with the NSIC value of not less than 65%. The powder coating of Example 6 was comparable to the powder coating of Example 4 in the quality of appearance but was remarkably superior in transportability and in blocking resistance when stored.

The powder coating of Example 5 and Example 6 having the standard deviation of particle diameter distribution not less than 20, formed a film equivalent to that of the conventional pigment containing powder coating in the quality of appearance.

The powder coating of Example 7 produced a film of superior appearance compared with the powder coating of Comparative Example 5 because the standard deviation of particle diameter distribution of the powder coating of Example 7 was not greater than 20 μm.

The powder coating of Example 8 produced a film of superior appearance compared with the powder coating of Comparative Example 6 because the standard deviation of particle diameter distribution of the powder coating of Example 8 was not greater than 20 μm.

The powder coatings of Examples 7 and 8 were different from the powder coatings of Examples 1-6 in the type of film-forming resin but because the standard deviation of particle diameter distribution was not greater than 20 μm, they produced films of improved appearance without compromise in transportability.

The powder coating of Example 7 having the standard deviation of particle diameter distribution not less than 20, formed a film equivalent to that of the conventional pigment containing acrylic powder coating in the quality of appearance. The powder coatings of Examples 9 and 10 had volume average particle diameters within the range of 20-50 μm with standard deviations of particle diameter distribution not exceeding 20 μm and, as such, produced films of superior appearance as compared with the powder coating of Comparative Example 7.

The powder coating of Example 10 in which the coating particles less than 700 μm in diameter accounted for not less than 40 weight % produced a film of still improved appearance as compared with the powder coating of Example 9.

[Effect of the Invention]

Having the construction described above, the powder coating of the present invention is improved in transportability and capable of yielding a film of very satisfactory appearance.

Claims

1. A powder coating comprising a film-forming resin having a volume average particle diameter of 20-50 μm with a standard deviation of particle diameter distribution not greater than 20 μm.

2. A powder coating comprising a film-forming resin having a volume average particle diameter of 20-50 μm with a standard deviation of particle diameter distribution not greater than 16 μm.

3. A powder coating comprising a film-forming resin having a volume average particle diameter of 20-50 μm with a standard deviation of particle diameter distribution not greater than 13 μm.

4. The powder coating as claimed in Claim 1, 2 or 3 wherein the particle diameter of the largest of particles constituting the powder coating is not greater than 90 μm.

5. The powder coating of Claim 4 wherein the particle diameter of the smallest of particles constituting the powder coating is not less than 1 μm.

6. The powder coating as claimed in Claim 1, 2 or 3 wherein said film-forming resin is at least one member selected from the group consisting of vinyl resin, polyethylene resin and polyamide resin.

7. The powder coating as claimed in Claim 1, 2 or 3 wherein said film-forming resin is at least one member selected from the group consisting of epoxy resin, thermosetting acrylic resin, and thermosetting polyester resin and which contains at least one of a curing agent and a curing accelerator.

8. The powder coating as claimed in Claim 7 which contains 5-80 parts by weight of said curing agent based on 100 parts by weight of said film-forming resin and 0.1-5 parts by weight of a curing accelerator based on 100 parts by weight of said film-forming resin.

9. The powder coating as claimed in Claim 1, 2 or 3 which contains at least one pigment selected from the group consisting of titanium dioxide, red iron oxide, yellow iron oxide, carbon black, phthalocyanine blue, phthalocyanine green and quinacridone red.

10. The powder coating as claimed in Claim 9 which contains 1-80 parts by weight of said pigment based on 100 parts by weight of the powder coating.

11. The powder coating as claimed in Claim 1, 2 or 3 wherein each of the particles constituting the powder coating comprises a composite of a first particle containing part of said film-forming resin and a second particle disposed on the surface of said first particle, said second particle containing the remainder of said film-forming resin and having a glass transition temperature of 50-150 °C.

12. The powder coating as claimed in Claim 11 wherein said second particle has a volume average particle diameter of 0.001-10 μm.

13. The powder coating as claimed in Claim 11 wherein the amount of said second particle is in a proportion of 0.05-35 parts by weight to 100 parts by weight of the powder coating.

14. The powder coating as claimed in Claim 11 wherein said second particle is made of at least one member selected from the group consisting of vinyl resin, acrylic resin, epoxy resin, polyester resin and melamine resin.

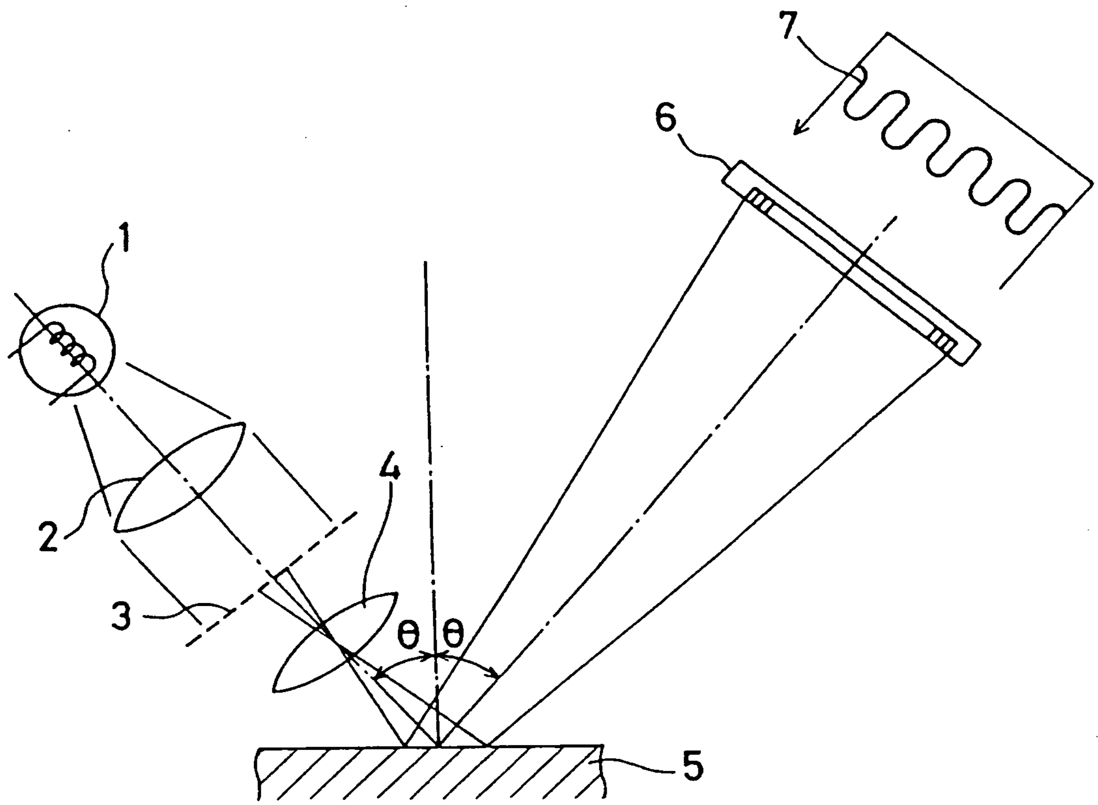

Fig. 1

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 95 10 8012

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
P,X	EP-A-0 653 466 (NIPPON PAINT CO) * page 3, line 52 - line 56; claims * ---	1-14	C09D5/03
P,X	EP-A-0 652 265 (NIPPON PAINT CO) * page 4, line 55 - page 5, line 3; claims * ---	1-14	
A	EP-A-0 536 791 (NIPPON PAINT CO) * claims * -----	1	
			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
			C09D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	22 September 1995	Deraedt, G

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document